

IMPROVED SEPARATORS FOR SILVER OXIDE-ZINC AND SILVER OXIDE-CADMIUM CELLS FOR SPACECRAFT APPLICATION

by
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and
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GPO PRICE \$ _____

CFSTI PRICE(S) \$ _____

Hard copy (HC) 1.00

Microfiche (MF) .50

ff 653 July 65



SECOND QUARTERLY PROGRESS REPORT
FEBRUARY 10, 1965 — MAY 9, 1965

prepared for
GODDARD SPACE FLIGHT CENTER
CONTRACT NAS 5-9107

THE BORDEN CHEMICAL COMPANY
Central Research Laboratory
Philadelphia, Penna. 19124

N66-13826
(ACCESSION NUMBER)
19
(PAGES)
CR 68847
(NASA CR OR TX OR AD NUMBER)

(THRU)
(CODE)
03
(CATEGORY)

FACILITY FORM 602

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PROJECT PERSONNEL

In carrying out the experimental work herein reported, the writers were assisted at The Borden Chemical Company by Assistants Ronald G. Burton and Charles J. Guinosso. At Burgess Battery Company, the sub-contract tests were under the supervision of Mr. Frederick A. Poss and Mr. J. R. Beckman.

SUMMARY & CONCLUSIONS

Improvement in the conductivity of methyl cellulose-polyacid films was brought about by increasing the proportion of the latter. These compositions while giving improved conductivity exhibited greater brittleness. 13826

Formulations incorporating polyvinyl alcohol or hydroxy ethyl cellulose were more flexible, but, as one would expect, less oxidation resistant.

Carboxymethyl methyl cellulose was prepared and made satisfactory films of good conductivity in 45% KOH. The presence of by-products and co-products was observed to contribute to the improved conductivity.

Work was continued on synthesizing polymers having requisite conductivity and flexibility without use of additives. To date none of these has proven entirely satisfactory. Author

I. ELECTRICAL RESISTANCE OF METHYL CELLULOSE-POLYACID MEMBRANES

The target of the formulations of polyacids and other highly polar polymers with methyl cellulose has heretofore been performance in 30% KOH. At this concentration several such combinations have shown sufficiently low resistance while at the same time exhibiting stability towards oxidation by dissolved silver oxide which was superior to that of cellophane, polyvinyl alcohol and hydroxyethyl polyvinyl alcohol.

The cell used for measuring the resistance of films was modelled after that of Shair (3), using a G. E. 65A impedance bridge and cathode ray oscilloscope to indicate null point. The finest measurement possible with this equipment is 0.5 ohm, so that with a 0.2 inch diameter opening the precision of measurement is at best 18 milliohms/square inch. Because of this relatively poor sensitivity many of the values for films measured in 30% KOH were substantially zero with fine differences being undetectable.

Electric Storage Battery Company and Yardney Electric Corporation have customarily used higher concentrations of 40-45% KOH at which concentration degradation of cellophane membranes is minimized. In their trials of membranes developed by us for 30% KOH (4)(5) they encountered and reported very high resistances on our C2, C3 and B3 films at concentrations over 40% KOH. They also found higher resistances on these films at the 30% level, using the more sensitive resistance equipment of the Salkind-Kelley design (6).

A program was accordingly set up to modify our formulations so as to lower the resistance of this class of films, particularly at the higher KOH concentrations. The solution to the problem is relatively simple and straightforward insofar as electrical resistance is concerned: that is, incorporate more of the polar constituents, the polyacids. However, since these components impart brittleness to the film it became necessary to study means of flexibilizing the new compositions. This was attempted by the use of monomeric plasticizers and by the use of compatible flexible polymers which included polyethylene oxide, polyvinyl alcohol and hydroxyethyl cellulose.

Exploratory formulations are given in Table I. All resistance measurements are made in 45% KOH at which concentration we could obtain approximate but significant differences in our resistance equipment. The criteria chosen for successful new candidates were: resistance below 100 milliohms/square inch and MIT flex at 200 grams tension above 500 cycles at 50% relative humidity.

The effect of plasticization was in general minor; in some cases the "plasticizer" showed embrittling effects. Incorporation of polyvinyl alcohol and hydroxyethyl cellulose showed some improvement in flexibility but it should be borne in mind that in these formulations conductive, more

flexible components are substituted in part for the brittle components so that no "synergistic" effect was exhibited. Several of those compositions were, however, chosen for trial in cell construction for cycle life tests. These included the following preparations:

	<u>Flex</u>	<u>Resistance in 45% KOH milliohms/in.²</u>
485-71 4 PVMAA/ 2 PVA1c/ 4 MC	1137	45
485-72 3 PVMAA/ 3 HEC/ 4 MC	456	19
485-73 3 PAA/ 3 HEC / 4 MC	1530	0

An unanticipated plasticization and reduction in electrical resistance was obtained in one series of experiments, as shown in the last four lines of the table. In order to obtain perfectly homogeneous solutions of methyl cellulose and polyacrylic acid it has been found that the pH must be raised to a minimum of 3.5. In exploring the effect of pH, increased flexibility appeared to be imparted by the addition of KOH as well as drastic reduction in resistance. This effect will be further explored in continuing work.

The stability towards silver oxide oxidation of the above "polymer-plasticized" films is given in Table II together with comparison data on related compositions.

Table I. - Methyl Cellulose Combinations with Polyacids and Additives

Type	Reference	Main Polymers (A)	Additives (B)	pH	Thick- ness mils	MIT Flex 200 g. cycles	Tensile psi	Resistance in 45% KOH milliohms/ in. ²
MC15	485-118	Meth.Cell. 15 cps	none	-	1.50	3937	11,000	11,600
C3	499-9	3PVMA/7MC	none	2.2	1.50	520	12,000	357
C4	445-102	4PVMA/6MC	none	2	1.50		3,720	169
C5	445-93	5PVMA/5MC	none	2	1.45		3,250	75
B3	421-113	3PAA/7MC	none	2.6	1.55	1319	11,700	350
B4	445-131	4PAA/6MC	KOH to	4.0	1.45	573	-	311
B5	445-141	5PAA/5MC	KOH to	4.0	1.35	1299	11,000	163
B5	445-145	5PAA/5MC	KOH,	4.0	1.40	800	9,940	458
			5% Polyox					
B5	445-144	5PAA/5MC	KOH,	4.0	1.35	868	-	82
			10% Polyox					
B4	445-132	4PAA/6MC	KOH to	5.0	1.45	537	-	35
B4	442-141	4PAA/6MC	KOH,	4.0	1.3	1189	-	1,885
			10% Polyox					
C4	445-137	4PVMA/6MC	KOH to	4.1	1.6	45	8,350	53
C4	445-138	4PVMA/6MC	KOH to	5.0	1.6	1	4,780	16
C4	445-139	4PVMA/6MC	KOH to	5.7	1.7	1	6,020	9
C5	445-140	5PVMA/5MC	none	2.2	1.45	263	13,700	66
C5	445-142	5PVMA/5MC	10% Polyox	2.2	1.55	202	12,400	82
C5	445-143	5PVMA/5MC	5% Polyox	2.2	1.50	238	14,100	7
C4	445-116	4PVMA/6MC	5% TEG	2.2	1.5	449	15,600	84
C4	482-1	"	KOH to	4.0	1.5	138	-	28
C4	482-2	"	KOH to	5.0	1.6	100	-	0
C4	482-3	"	KOH to	6.0	1.6	234	-	16
C5	482-8	5PVMA/5MC	10% PValc(K)-		1.45	524	-	poor film
C5	485-40	5PVMA/5MC	20% PValc(L)-		1.50	1137	14,600	69
C5	485-29	5PVMA/5MC	3 TEG	-	1.5	827	15,000	53
			17% PValc(L)					
B5	485-64A	5PAA/5MC	5% PValc(L)4.(M)		1.5	600	-	3
B5	-64B	"	10% PValc(L)4.(M)		1.5	590	-	0
B5	-64C	"	22% PValc(L)4.(M)		1.6	506	-	3
B5	485-33A	"	30% Polyox	5	1.5	39	4,280	0
			(O)					
B5	-33B	"	20% Polyox	5	1.5	211	5,190	3
			(O)					
B5	-33C	"	10% Polyox	5	1.6	524	7,030	0
			(O)					
F5	485-55	5PMA/5MC15	KOH to	5	1.4	78	11,200	3
F5	-56	"	KOH to	6	1.5	too	brittle	to test
F5	-59	"	KOH to	4.5	1.5	395	-	3
	485-26A	5PVMA/3HEC/ 2MC15		-	1.4	353	13,400	9
	485-26B	5PVMA/2HEC/ 3MC15		-	1.4	148	14,100	66
	485-26C	4PVMA/2HEC/ 4MC15		-	1.5	552	15,500	63
	485-26D	3PVMA/3HEC/ 4MC15		-	1.4	897	13,500	53

(CONTINUED ON FOLLOWING SHEET)

Table I - Methyl Cellulose Combinations with Polyacids and Additives (CONTINUED)

Type	Reference	Main Polymer (4)	Additives (B)	pH	Thick- ness mils	MIT Flex 200 g. cycles	Tensile psi	Resistance in 45% KOH milliohms/ in. ²
	445-146	Ethulose	none	-	1.2	10,000	11,000	620
	485-19	2PVMMMA/8	none	-	1.5	2,845	12,600	201
	485-21	Ethulose 3PVMMMA/7	none	-	1.5	928	13,400	226
	485-34	Ethulose 4PVMMMA/6	none	-	1.7	62	11,000	69
	485-35	Ethulose 5PVMMMA/5	none	-	1.9	21	8,900	53
B5	485-76	Ethulose 5PAA/5MC15	KOH to	3.5	1.5	217	-	22
B5	485-78A	"	5% PValc(L)	3.5(N)	1.5	216	14,500	12
B5	485-78B	"	10% PValc(L)	3.5	1.6	507	16,100	6
B5	485-78C	"	20% PValc(L)	3.5	1.5	796	14,400	20
C5	485-71	5PVMMMA/5MC15	25% PValc(LO	-	1.5	836	15,900	44
	-72	3PVMMMA/3HEC/ 4MC15		-	1.5	456	15,100	19
B3	421-113	3PAA/7MC	none	2.6	1.6	1,032	-	283
B3	445-123	3PAA/7MC	+ 3.4% KOH	4.0	1.4	1,622	-	235
B3	445-124	3PAA/7MC	+ 10.8% KOH	5.0	1.4	1,181	-	31
B3	445-125	3PAA/7MC	+ 18.7% KOH	6.0	1.6	5,547	-	0

NOTES:

PAA Polyacrylic acid Acrysol A5 (Rohm & Haas)

PMMA Polymethacrylic Acid (Borden Prepn. 436-6)

PVMMMA Poly(vinyl methylether-maleic anhydride) Gantrez (General Aniline)

HEC Hydroxyethyl Cellulose QP09 (Union Carbide)

Ethulose is a cellulose derivative containing ethyl groups (d.s. 0.9) and hydroxyethyl groups (d.s. 0.8) (Chemaster Corp.)

T.E.G. Tetraethylene glycol (Dow)

(B) Percents are based on weight added per 100 parts A

(D) Film not insoluble in 30% KOH (disintegrated)

(K) 30-98 Lemol (completely hydrolyzed polyvinyl alcohol) (Borden)

(L) 22-88 Lemol (partially hydrolyzed polyvinyl alcohol) (Borden)

(M) B5 was adjusted to pH 4 with KOH

(N) B5 was adjusted to pH 3.5 with KOH

(O) Films not homogeneous. Microscope showed dispersed phase.

Polyox is polyethylene oxide WSRN80 (Union Carbide)

Table II. - Oxidation Resistance of Representative Films
(72 hours continuous stirring in KOH saturaged with Ag₂O)

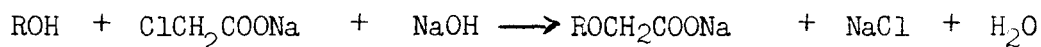
No.	Reference	Composition	Ratio of Strength after 72 hrs oxidation/ "orig" strength			Resistance in 45% KOH milliohms/ in. ²
			30% KOH	40% KOH	45% KOH	
7	485-72	3 PVMA/3 HEC/4 MC	.91	-	.90	19
6	485-71	4 PVMA/2 PValc/4 MC	.65	-	.94	45
8	485-109	2 Ethulose/8 MC	.64	-	.92	185
9	485-73	3PAA/3 HEC/4 MC	.88	-	.95	107
	421-47	HEC	sol.	-	.95	334
	445-108	PValc(30-98)	.76	.91	.89	6
	485-13	Ethulose	-	-	1.03	620
		PUDO Cellophane	.15	.61	.83	31
2	485-104	C3 (reference)	.86	.81 ⁹⁷	.97	357

"Original" strength is defined as the strength of the film after 72 hours stirring in the KOH in the absence of Ag₂O.

II. CARBOXYMETHYL METHYL CELLULOSE

Commercial methyl cellulose has a degree of substitution of about 1.8 methoxy groups per anhydroglucose unit, leaving 1.2 hydroxyls unsubstituted. Reaction of the residual hydroxyls with sodium chloroacetate would give the highly polar $-\text{OCH}_2\text{COONa}$ group which might be expected to give enhanced conductivity over the original methyl cellulose. Such a product might present an advantage over two component mixtures of methyl cellulose and polyacids.

Several preparations were carried out in which methyl cellulose was reacted with sodium chloroacetate and sodium hydroxide. The procedures were modifications of those employed in the carboxymethylation of cellulose to produce carboxymethyl cellulose (1), (2). Thus where R represents methyl cellulose:



As judged by exotherms, consumption of NaOH, the appearance of ionic chlorine and pronounced solubility changes, the reaction occurred readily. Procedures appropriate to the purification of carboxymethyl cellulose did not result in salt-free products. The preparations were used, nevertheless, to cast film and study film properties. Besides residual NaCl, unreacted sodium chloroacetate and sodium hydroxide, sodium glycollate is a possible co-product. The effect of these monomeric products on electrical resistance was explored by their addition to methyl cellulose, as shown in Table III.

From the table it can be seen that the carboxymethyl methyl cellulose films showed greatly lowered resistance as compared to methyl cellulose, in two cases around the usual values for cellophane and polyvinyl alcohol (below 50 milliohms/sq.in.). At the same time it should be noted that all the monomeric additives lowered resistance so that their presence was undoubtedly a contributing factor.

The effect of monomeric additives on electrical resistance will be further studied.

Table III. - Films from Carboxymethyl Methyl Cellulose and Related Compositions

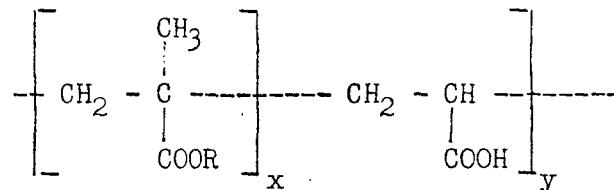
Reference	Composition	MIT Flex* Thick- ness mils cycles		Resistance 45% KOH milliohms/ in. ²	Na %
421-86	Methyl Cellulose (Methocel 15)	1.5	10,000	7,250	-
483-5	Carboxymethyl Methyl Cellulose (1)	1.5	5,717	258	2.2
483-7	" " " (1)	1.6	4,513	9	9.7
482-23	" " " (2)	1.4	953	3	15.1
485-79	MC + 25% NaCl	1.4	-	283	-
504-132	MC + 3% NaOH	1.5	-	1,655	-
504-133	MC + 6% NaOH	1.5	4,465	330	-
504-134	MC + 10% NaOH	1.5	-	0	-
485-101A	MC + 2.5% Glycolic Acid	1.6	-	4,440	-
485-101B	MC + 5.0% " "	1.6	-	2,000	-
485-101C	MC + 10% " "	1.5	-	1,350	-
485-100A	MC + 5% Sod. Glycolate	1.5	-	200	-
485-80	MC + 25% " "	1.5	-	94	-

* Under 200 g tension.

- (1) Reaction was carried out at room temperature by mixing an aqueous dough of methyl cellulose, NaOH and sodium chloroacetate. See reference (1)
 (2) Reaction was carried in an isopropanol suspension. See reference (2).

III. METHACRYLATE-ACRYLATE POLYMERS

In the First Quarterly Report under this contract, a procedure was described whereby a methacrylate ester monomer was copolymerized with an acrylate ester monomer and the resulting copolymer subjected to alkaline hydrolysis of the acrylate moiety. This was followed by precipitation of the resulting hydrolyzate into hydrochloric acid to give a copolymer with acid groups of the general structure:



Several products so prepared appeared to have the required low resistance for separators and showed the following solubility properties: insoluble in water, soluble 2% KOH, insoluble in 30% and 40% KOH. However, all were too brittle for unsupported films, and membranes had to be constructed by impregnation of 1.5 mil Dynel cloth with dioxane solutions of the polymer.

It was believed, judging from the semi-liquid form of the unhydrolyzed polymers and the low viscosity of the solutions, that molecular weights were too low, a result of the chain stopping effect of the ketone solvents in which the polymerizations were conducted. In continuation of this approach benzene was used as solvent at relatively low solvent concentration, 50% as compared to 10% with the ketonic solvents. The resulting polymers in their ester form were solids ranging from soft through flexible to hard flexible films. When converted to acid form, however, brittle films of high resistance were generally found. One of these, 482-28, Table IV, developed further lowered resistance when the film itself was heated several days in 45% KOH at 50° C. This indicated that the preparative saponification step had not been complete. An attempt to carry out the hydrolysis with mineral acid was unsuccessful (482-58).

In order to obviate the difficult and laborious hydrolysis step a number of copolymerizations of methacrylate esters with acidic monomers was carried out by emulsion polymerization. All the products gave continuous but brittle films. An attempt to plasticize by adjusting the pH upward with aminopropanol showed either no plasticization at pH's below 4 or gave weak waxy films at higher pH's. See Tables V, VI.

A series of bulk polymerizations was carried out using butyl methacrylate and maleic anhydride or itaconic acid. Nominal amounts of methanol were used to insure initial compatibility of the reactants. Some crosslinking occurred in some of the preparations. Although flexible films were obtained from some of the formulations, resistance in 45% KOH was too high in all cases. Table VII.

Table IV. - Solution Polymerization in Benzene, Methanol

Reference		445- 17	445- 37	482- 20	482- 26	485- 103	485- 69	485- 105	482- 28	482- 58	482- 75	482- 77
Methyl Acrylate												
	g.	28	28	28	43	62	76	52	43	43	-	-
	M	.32	.32	.32	.50	.72	.88	.60	.50	.50	-	-
Itaconic Acid												
	g.	-	-	-	-	-	-	-	-	-	52	26
	M	-	-	-	-	-	-	-	-	-	.40	.20
Methacrylate Ester												
Octyl (A)	g.	94	94	94	-	-	-	-	-	-	-	-
	M	.48	.48	.48	-	-	-	-	-	-	-	-
Heptyl	g.	-	-	-	.92	-	-	-	-	-	-	-
	M	-	-	-	.50	-	-	-	-	-	-	-
Butyl	g.	-	-	-	-	-	-	-	72	72	85	114
	M	-	-	-	-	-	-	-	.50	.50	.60	.80
Methyl	g.	-	-	-	-	108	90	40	-	-	-	-
	M	-	-	-	-	1.08	.90	.40	-	-	-	-
Product Film												
MIT Flex (C)		-	-	v. soft	v. soft	flex- ible	flex- ible	1	v. soft	v. soft	-	-
Prod. Acid Form												
MIT Flex (C)		1	1	flex	1	-	(E)	-	1	(D)	flakes	flakes
Prod. Acid Form												
Resistance (B)		2.6×10^4	9×10^4	6×10^4	1.1×10^3	-	(E)	-	7.2×10^3	-	-	-
Solvent												
		C_6H_6	C_6H_6	C_6H_6	C_6H_6	C_6H_6	C_6H_6	C_6H_6	C_6H_6	C_6H_6	MeOH	MeOH

Procedure: Polymerizations were carried out in approx. 50 % benzene using .1 to .2 grams azobisisobutyronitrile as initiator. Temperature 80° C. Time 4-16 hours until solids content indicated completion of reaction. Benzene was then replaced with isopropanol and film cast. Where film was prepared in acid form the product was saponified with KOH and precipitated into excess HCl. This was then filtered, dried, taken up in isopropanol and cast as film. See page 17, First Quarterly Report NAS 5-9107 Nov. 10, 1964 - Feb. 9, 1965.

NOTES FOR THIS TABLE ON FOLLOWING SHEET.

Notes for Table IV:

- (A) Octyl - 2-ethylhexyl
- (B) Resistance in 45% KOH milliohms/sq.in. film.
- (C) Cycles to break under 200 g. tension.
- (D) Tried to hydrolyze with HCl. No reaction.
- (E) The 485-63 product as co-polymer ester in film form was hydrolyzed in 45% KOH at 37° C. Resistance readings vs. time are shown below:

overnight at room temperature	1.9×10^5	milliohms/sq.in.
after 6 days at 37° C.	2.0×10^3	" "
after 12 days at 37° C.	disintegrated	

- (F) Film 482-28

after 5 days at 50° C.	9	milliohms/in.sq.
after 19 days at 50° C.	12	" "

Film was still intact. This shows original film was not completely hydrolyzed. The originally hydrolyzed film was, however, very brittle.

Table V. - Emulsion Polymerization Butyl Methacrylate/Acrylic Acids

Prep.	485-128		485-140		485-146		485-134		499-1		499-82		499-97	
	g.	M	g.	M	g.	M	g.	M	g.	M	g.	M	g.	M
Butyl Meth- acrylate (B)	36	.25	36	.25	49.7	.35	36	.25	36	.25	36	.25	114	.80
Acrylic Acid (A)	18	.25	18	.25	14.4	.20	-	-	-	-	18	.25	14.4	.20
Methacrylic Acid (A)	-	-	-	-	-	-	21	.25	21	.25	-	-	-	-
H ₂ O ₂ 35%	-	-	1.2	-	1.2	-	-	-	1.2	-	1.2	-	1.2	-
Ascorbic Acid 5%	-	-	4.7	-	4.7	-	-	-	4.7	-	4.7	-	4.7	-
Am. Persulfate 5%	1.2	-	-	-	-	-	4.8	-	-	-	-	-	-	-
Sod. Metabi- sulfite 5%	1.4	-	-	-	-	-	9.8	-	-	-	-	-	-	-
NH ₄ OH 28%	-	-	-	-	-	-	-	-	-	-	20	-	-	-
Crud Approx.%	1.0		0.2		0.2		75.		99		0.2			
Solids, %	12.4		10.3		10.6		-		-		11.0		24.9	
Solids calcd. %	11.0		10.3		12.4		-		-		11.3		22.2	
Film description	brittle		brittle		brittle		brittle		brittle		powder		brittle	
KOH Soly. 30%	disintegrated				disintegrated				disintegrated				disintegrated	
KOH Soly. 40%	"				"				"				"	

Polymerizations were carried out in all glass resin flasks under nitrogen at room temperature at about 10% concentration of monomer in water, using lauryl sulfate (0.1 part) as emulsifying agent. Oxidant was added followed by incremental addition of reductant over a period of one hour. A post-heating of one hour at 50° C. followed. Films were cast directly from the emulsion.

Table VI - Emulsion Polymerization Butyl Methacrylate with Acidic Monomers

Prep.	495-79		499-85		499-91		499-119	
	g.	M	g.	M	g.	M	g.	M
Butyl Methacrylate	36	.25	36	.25	36	.25	50	.35
Maleic Anhydride	24.5	.25	24.5	.25	-	-	-	-
Itaconic Acid	-	-	-	-	33	.25	-	-
Acrylonitrile	-	-	-	-	-	-	8	.15
H ₂ O ₂ (35%)	1.2		1.2		1.2		1.2	
Ascorbic Acid (5%)	4.7		4.7		4.7		4.7	
NH ₄ OH (28%)	-		20.0		0		0	
Water	450		450		450		450	
Sod. Lauryl Sulfate (25%)	2.2		2.2		2.2		2.2	
Crud - approx.	high		high		-		low	
Solids %	12.5		9.3		-		11.3	
Film Description	flakes		powder		flakes		v. brittle	

Table VII. Bulk Polymerizations. Butyl Methacrylate Co-polymers With Acidic Monomers

Ref. 499-	Method	Butyl Meth- acryl- ate g.	Maleic Anhy- dride g.	Mono- butyl Ita- conate g.	Meth- anol g.	BMA/Acid mol ratio	BzO ₂ g.	AIBN g.	Benz- il g.	Film Sol- vent	Proper- ties Descrip- tion	Resist- ance in 45% KOH milli- ohms/in. ²
99	Light, 8 days	22.2	13.6	-	4.2	.158/.133	.05	-	.05	MEK	Too soft	1.97x10 ⁴
100	" "	22.2	13.6	-	4.2	.158/.139	-	.05	.05	MEK	Too soft	9.17x10 ³
101	" "	26.7	10.2	-	3.2	.188/.104	.05	-	.05	Insol. in all solvent	" " "	-
102	" "	31.1	6.8	-	2.2	.219/.069	.05	-	.05	"	" " "	-
103	" "	35.4	3.4	-	1.0	.250/.035	.05	-	.05	"	" " "	-
104	" "	28.0	-	12.0	1.0	.197/.065	.05	-	.05	"	" " "	-
107	" "	22.2	13.6	-	4.2	.158/.139	-	-	.05	isoPrOH	Too soft	-
108	" "	22.2	13.6	-	4.2	.158/.139	-	-	-	MEK	Too soft	-
109	" "	26.7	10.2	-	3.2	.188/.104	-	-	.05	isoPrOH	" "	-
110	" "	31.1	6.8	-	2.1	.219/.069	-	-	.05	"	Flexible soft	2.4x10 ⁴
111	" "	35.4	3.4	-	1.0	.250/.035	-	-	.05	"	Flexible	1.7x10 ⁴
112	" "	28.0	2	12.0	1.8	.197/.065	-	-	.05	"	Too brittle	3.7x10 ⁴
113	Heat 27 hrs. 50°C	22.2	13.6	-	4.2	.158/.139	.05	-	-	"	Flexible, soft	7.6x10 ³
114	" "	22.2	13.6	-	4.2	.158/.137	-	.05	-	"	Very soft	8.6x10 ⁵
115	" "	26.7	10.2	-	3.2	.188/.104	.05	-	-	"	Very soft	1.2x10 ⁶
116	" "	31.1	6.8	-	2.1	.219/.069	.05	-	-	"	Flexible, soft	2.3x10 ²
117	" "	35.4	3.4	-	1.0	.250/.035	.05	-	-	"	Flexible	1.2x10 ⁵
118	" "	28.0	-	12.0	1.0	.197/.065	.05	-	-	"	Brittle	1.8x10 ⁵

Polymerizations were carried out in sealed 2 oz. bottles under nitrogen in light or by heat as indicated. At the end of the polymerization period the bottles were broken, the solid polymers removed, dissolved in appropriate solvent and cast as films.

AIBN - azobisisobutyronitrile

BzO₂ - benzoyl peroxide

References;

- (1) McLaughlin, Can. J. Res. 28B, p. 731
- (2) Klug, U. S. 2,517,577
- (3) Shair et al, Ind. Eng. Chem. 48, 381-5 (1956).
- (4) Fourth Quarterly Report, Yardney, DA 36-039-AMC02238E
- (5) Private communication.
- (6) Cooper-Fleischer, Characteristics of Separators for Alkaline Silver Oxide Zinc Secondary Batteries. Screening Methods. AF Aero Propulsion Laboratory, p. 69